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PATENT SPECIFICATION

751.129



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COMPLETE SPECIFICATION

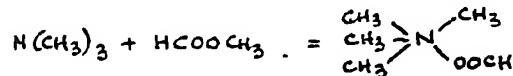
Improvements in the Production of Quaternary Ammonium Salts

We, BADISCHE ANILIN- & SODA-FABRIK AKTIENGESELLSCHAFT, a Joint Stock Company recognised under the laws of Germany, of Ludwigshafen on Rhein, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 We have found that quaternary ammonium salts of carbonic acid or formic acid can be obtained in a very advantageous manner by heating to reaction tertiary amines of which the nitrogen atom is not a member of a ring of aromatic character with esters of the said acids.

20 Examples of tertiary amines of which the nitrogen atom is not a member of a ring of aromatic character are trialkylamines, such as trimethylamine, triethylamine, dimethyl-ethanolamine, diethyl-i-propylamine, dimethyloleylamine. Tertiary cycloaliphatic, aromatic or saturated heterocyclic amines may also be mentioned, such as N,N-diethyl-cyclohexylamine, N,N-dimethylaniline or 1,6-dipyrrolidino-hexane. Suitable esters of carbonic or formic acid are for example the alkyl, cycloalkyl and aryl esters, such as methyl formate and carbonate, isopropyl, lauryl and oleyl formate and carbonate, and also cyclohexyl and phenyl formate and carbonate.

30 For example tetramethyl ammonium formate is formed by the reaction of trimethylamine with methyl formate according to the following equation:



40 The reaction is carried out for example by heating the tertiary amine with about the calculated amount of the ester, preferably under pressure, to temperatures above 100° C., for example between 120° and 250° C. The temperature and the duration of the heating

[Price 3s. 0d.]

depend on the reactivity of the ester and amine used. In many cases the employment of solvents or diluents is advantageous. Tetramethyl ammonium formate is a colourless crystalline mass and is formed in very good yields.

45 The following Examples will further illustrate this invention but the invention is not restricted to these Examples. The parts are parts by weight.

EXAMPLE 1.

50 250 parts of methyl formate and 270 parts of trimethylamine are heated at 180 to 200° C. in an autoclave for 12 hours. 370 parts of tetramethyl ammonium formate are thus obtained. It is obtained in pure form by recrystallization from a mixture of isopropanol and acetone.

EXAMPLE 2.

55 350 parts of 1,6-di-(dimethylamino)-hexane and 200 parts of dimethyl carbonate are heated at 105° to 110° C. in a stirring vessel for 12 hours. The 1,6-hexamethyl-diammonium carbonate (500 parts) formed thereby separates in solid form. By reaction with equimolecular amounts of acid the corresponding 1,6-hexamethyl-diammonium salts of other acids are obtained with the splitting off of carbon dioxide, for example 1,6-hexamethyl-diammonium tartrate having the melting point 169° C.

EXAMPLE 3.

60 44 parts of glycol carbonate together with 60 parts of trimethylamine in 80 parts of methanol are heated for 6 hours at 100° C. in an autoclave. After cooling, 28 parts of trimethylamine are recovered. After distilling off the methanol, 72 parts of the condensation product which is insoluble in ether remain behind. If this is reacted with an inorganic or organic acid, the corresponding choline salt is obtained with the splitting off of carbon dioxide. Thus for example, if the condensation product obtained above is reacted with 75 parts of tartaric acid, 125 parts of choline bitartrate are obtained which melts at 145° C. after recrystallization from methanol.

Price 4s 6d

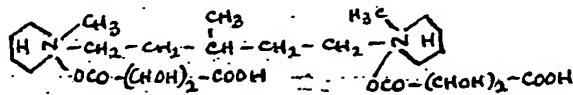
Price 5s

EXAMPLE 4.

5 A solution of 80 parts of 3-methyl-1,5-dipyrrolidinopentane in 110 parts of methanol and 65 parts of dimethyl carbonate is heated to from 90° to 100° C. in an autoclave for 10 hours. The contents of the autoclave are combined with an aqueous suspension of

crystallized tartaric acid until the evolution of carbon dioxide has ceased and the reaction has become distinctly acid. After filtering off 10 any undissolved tartaric acid, the reaction product is precipitated with acetone. The precipitate of the tartrate of 3-methyl-1,5-di-(methylpyrrolidinium)-pentane.

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which is first formed in an oily form crystallizes on standing for some time. It is recrystallized from five times its quantity of methanol (m.p. = 210° C.).

25 When using 1,6-dipyrrolidinohexane instead of and in about the same quantity as 3-methyl-1,5-dipyrrolidinopentane and proceeding in an analogous manner as in the preceding paragraph, the tartrate of 1,6-di-(methylpyrrolidinium)-hexane is obtained.

What we claim is:—

1. A process for the production of quaternary ammonium salts of carbonic acid and

30 formic acid, which consists in heating a tertiary amine with an ester of carbonic acid or formic acid until reaction occurs, the nitrogen atom of said tertiary amine not being a member of a ring of aromatic character.

2. The process for the production of quaternary ammonium salts substantially as described in any of the foregoing Examples.

3. Quaternary ammonium salts when obtained by the process claimed in claim 1 or 2.

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